

Suppression of phase separation and giant enhancement of superconducting transition temperature in $\text{FeSe}_{1-x}\text{Te}_x$ thin films

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We demonstrate the successful fabrication on CaF_2 substrates of $\text{FeSe}_{1-x}\text{Te}_x$ films with $0 \leq x \leq 1$, including the region of $0.1 \leq x \leq 0.4$, which is well known to be the “phase-separation region”, via pulsed laser deposition which is a thermodynamically non-equilibrium method. In the resulting films, we observe a giant enhancement of the superconducting transition temperature, T_c , in the region of $0.1 \leq x \leq 0.4$: the maximum value reaches 23 K, which is approximately 1.5 times as large as the values reported for bulk samples of $\text{FeSe}_{1-x}\text{Te}_x$. We present a complete phase diagram of $\text{FeSe}_{1-x}\text{Te}_x$ films. Surprisingly, a sudden suppression of T_c is observed at $0.1 < x < 0.2$, while T_c increases with decreasing x for $0.2 \leq x < 1$. Namely, there is a clear difference between superconductivity realized in $x = 0 - 0.1$ and in $x \geq 0.2$. To obtain a film of $\text{FeSe}_{1-x}\text{Te}_x$ with high T_c , the controls of the Te content x and the in-plane lattice strain are found to be key factors.

$\text{FeSe}_{1-x}\text{Te}_x$ | thin film growth | compressive strain | complete phase diagram

Abbreviations: PLD, pulsed laser deposition; XRD, X-ray diffraction

Since the discovery of superconductivity in $\text{LaFeAs}(\text{O},\text{F})$ [1], many studies concerning iron-based superconductors have been conducted. FeSe is the iron-based superconductor with the simplest crystal structure[2]. The T_c of FeSe is approximately 8 K, which is not very high in comparison with other iron-based superconductors. However, the value of T_c strongly depends on the applied pressure, and the temperature at which the resistivity becomes zero, T_c^{zero} , reaches as high as ~ 30 K at 6 GPa[3]. This suggests that FeSe samples with higher T_c are available by the fabrication of thin films because we can introduce lattice strain. Indeed, we have previously reported that FeSe films fabricated on CaF_2 substrates exhibit T_c values approximately 1.5 times higher than those of bulk samples because of in-plane compressive strain[4]. On the other hand, superconductivity with T_c of 65 K has recently been reported in a monolayer FeSe film on SrTiO_3 [5, 6]. It is unclear whether this superconductivity results from the characteristics of the interface. However, this finding indicates that FeSe demonstrates potential as a very-high- T_c superconductor.

The partial substitution of Te for Se in FeSe also raises T_c to a maximum of 14 K at $x = 0.5 - 0.6$ [7]. In $\text{FeSe}_{1-x}\text{Te}_x$, it is well known that we cannot obtain single-phase samples with $0.1 < x < 0.4$ because of phase separation[7]. Here, we focus on this region of phase separation. Generally, the process of film deposition involves crystal growth in a thermodynamically non-equilibrium state. Thus, film deposition provides an avenue for the synthesis of a material with a metastable phase. In this letter, we report the fabrication of epitaxial thin films of $\text{FeSe}_{1-x}\text{Te}_x$ with $0 \leq x \leq 1$ on CaF_2 substrates using the pulsed laser deposition (PLD) method. We demonstrate that single-phase epitaxial films of $\text{FeSe}_{1-x}\text{Te}_x$ with $0.1 \leq x \leq 0.4$ are successfully obtained and that the maximum value of T_c is as large as 23 K, which is higher than the previously-reported values for bulk and film samples of $\text{FeSe}_{1-x}\text{Te}_x$ [7, 8, 9, 10, 11, 12], except for those of the mono-

layer FeSe films[5, 6]. Our results clearly show that the optimal Te content for the highest T_c for $\text{FeSe}_{1-x}\text{Te}_x$ films on CaF_2 is different from the widely-believed value for this system.

Figure 1a presents the X-ray diffraction patterns of $\text{FeSe}_{1-x}\text{Te}_x$ films for $x = 0 - 0.5$ on CaF_2 . Here and hereafter, the Te content x of our films represents the nominal Te composition of the polycrystalline target. With the exception of an unidentified peak in the $\text{FeSe}_{0.5}\text{Te}_{0.5}$ film, only the 00 l reflections of a tetragonal PbO -type structure are observed, which indicates that these films are well oriented along the c axis. Figures 1b and 1c present enlarged segments of these plots near the 001 and 003 reflections, respectively. The 2θ values of the peak positions decrease with increasing x in a continuous manner, which is consistent with the fact that the c -axis length increases with increasing x . It should be noted that the values of the full widths at half maximum (FWHM), $\delta(2\theta)$, of the $\text{FeSe}_{1-x}\text{Te}_x$ films with $x = 0.1 - 0.4$, which is known as the region of phase separation in the bulk samples[7], are $\delta(2\theta) = 0.2 - 0.3^\circ$ for the 001 reflection and $\delta(2\theta) = 0.4 - 0.6^\circ$ for the 003 reflection, which are nearly the same as the values for the FeSe and $\text{FeSe}_{0.5}\text{Te}_{0.5}$ films. This result is in sharp contrast to the previously-reported result that the FWHM was broad *only* in films of $\text{FeSe}_{1-x}\text{Te}_x$ with $x = 0.1$ and 0.3 ,[13] where phase separation has been believed to occur. The results presented in Figs. 1a-1c indicate the formation of a single phase in our $\text{FeSe}_{1-x}\text{Te}_x$ films with $x = 0.1 - 0.4$.

In Figure 1d, the c -axis lengths of 29 films of $\text{FeSe}_{1-x}\text{Te}_x$ are plotted as a function of x . The values of the c -axis lengths vary

Significance

To clarify the mechanism of superconductivity in iron-based superconductors, it is crucial to investigate $\text{FeSe}_{1-x}\text{Te}_x$, which has the simplest crystal structure among them. There is, however, a serious obstacle to the understanding of its superconductivity; phase separation occurs in the region of $0.1 \leq x \leq 0.4$, and thus a whole phase diagram has not been available. Here we report the successful fabrication of $\text{FeSe}_{1-x}\text{Te}_x$ films with $0 \leq x \leq 1$. This is the first demonstration of the suppression of the phase separation. What is more notable is that a giant enhancement of T_c is observed in the “phase-separation region”. The complete phase diagram that we present provides a novel perspective for the mechanism of superconductivity of this material.

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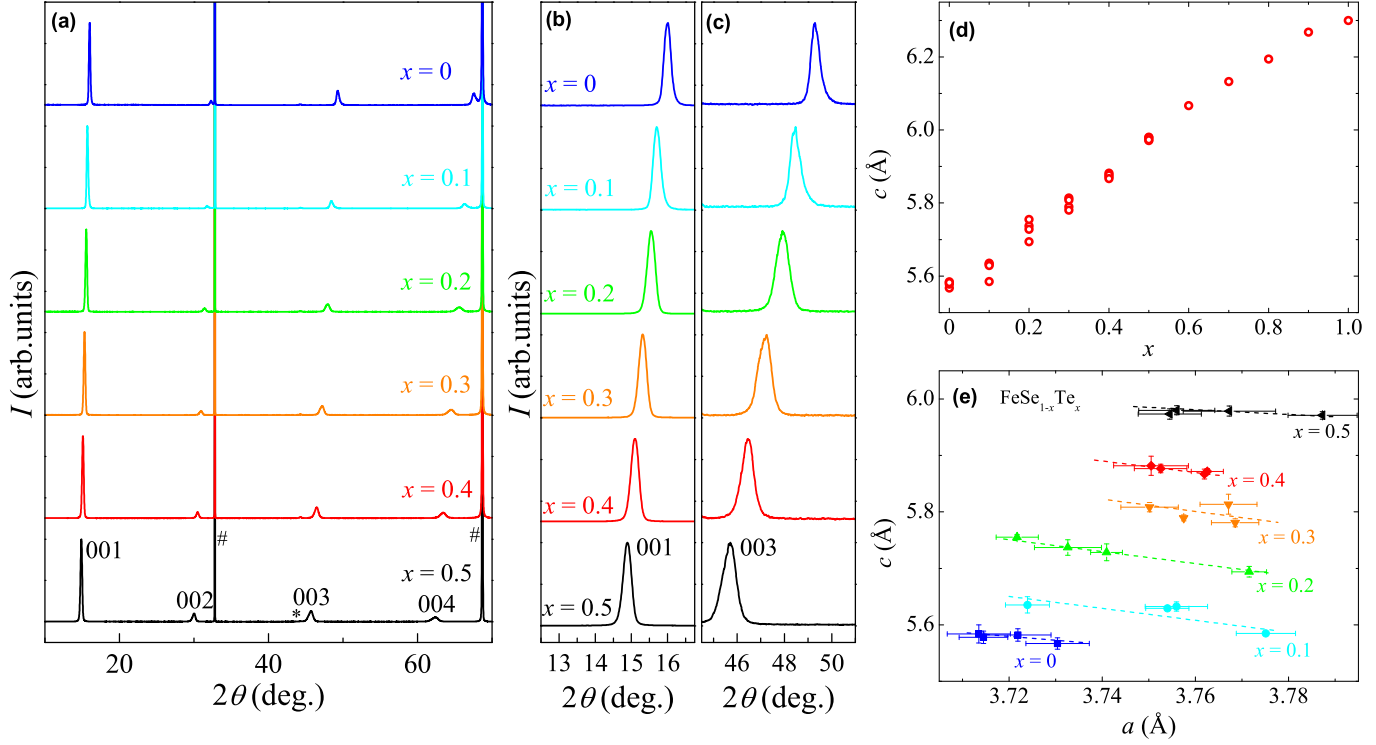


Fig. 1. (a) Out-of-plane X-ray diffraction patterns of $\text{FeSe}_{1-x}\text{Te}_x$ thin films for $x = 0 - 0.5$ with film thicknesses of 120-147 nm. The number signs represent peaks associated with the substrate. The asterisk represents an unidentified peak. Enlarged segments of the plots presented in (a) near the 001 and 003 peaks are shown in (b) and (c), respectively. (d) The c -axis lengths of $\text{FeSe}_{1-x}\text{Te}_x$ films, where the x value indicated on the horizontal axis is the nominal Te content. (e) Relations between the a -axis and c -axis lengths in $\text{FeSe}_{1-x}\text{Te}_x$ films. The colors and shapes of the symbols correspond to the Te content x , as shown in the figure. The dashed lines are guides for the eye. The data for $x = 0$ and 0.5 presented in (a)-(e) are cited from our previous papers[4, 14].

almost linearly with the nominal Te contents of the targets in the whole range of x including both end-member materials. The evident formation of a single phase and the systematic change in the c -axis length strongly indicate that the nominal Te content of the polycrystalline target is nearly identical to that of the final $\text{FeSe}_{1-x}\text{Te}_x$ film. Note that the compositional analysis of grown films using scanning electron microscopy/energy-dispersive X-ray (SEM/EDX) analysis is impossible for $\text{FeSe}_{1-x}\text{Te}_x$ films on CaF_2 substrates because the energies of the K-edge of Ca and the L-edge of Te are very close to each other. The above-mentioned features indicate that phase separation is suppressed in our $\text{FeSe}_{1-x}\text{Te}_x$ films with $x = 0.1 - 0.4$ on CaF_2 substrates. To our knowledge, this result is the first manifestation of the suppression of phase separation in $\text{FeSe}_{1-x}\text{Te}_x$ with $x = 0.1 - 0.4$.

Figure 1e presents the relations between the a -axis and c -axis lengths in films of $\text{FeSe}_{1-x}\text{Te}_x$. At first glance, there seems to be no relations between the a -axis length and x , in sharp contrast to the behavior of the c -axis length. The a -axis and c -axis lengths of films with the same x show a weak negative correlation. This behavior cannot be explained by a difference in Te content of a film, which should result in a positive correlation. By contrast, if variations in c are caused by a difference in in-plane lattice strain, this behavior can be explained in terms of the Poisson effect. Indeed, the a -axis lengths of films of FeSe and $\text{FeSe}_{0.5}\text{Te}_{0.5}$ are smaller than those of bulk samples with the same composition. Thus, we consider that the a -axis length predominantly depends on the in-plane lattice strain rather than the Te content x . One might think that this behavior looks strange, because the lattice constant

of CaF_2 ($a_{\text{CaF}_2}/\sqrt{2}$) is longer than the a of $\text{FeSe}_{1-x}\text{Te}_x$, which usually leads to a tensile strain. In the previous paper, the penetration of F^- ions from the CaF_2 substrates into the films has been proposed as a possible mechanism for nontrivial compressive strain in $\text{FeSe}_{1-x}\text{Te}_x$ films on CaF_2 substrates[15]. Because of smaller ionic radius of F^- than that of Se^{2-} , this peculiar compressive strain can be explained by the partial substitution of F^- for Se^{2-} near the interface between a film and a substrate.

Figures 2a-2d present the temperature dependences of the electrical resistivities, ρ , of 16 films of $\text{FeSe}_{1-x}\text{Te}_x$ for $x = 0.1 - 0.4$. The value of T_c depends on the film thickness, even in films with the same x . The highest T_c^{onset} , which is defined as the temperature where the electrical resistivity deviates from the normal-state behavior, and the T_c^{zero} of the $\text{FeSe}_{1-x}\text{Te}_x$ films are 13.2 K and 11.5 K, respectively, for $x = 0.1$; 22.8 K and 20.5 K, respectively, for $x = 0.2$; 20.9 K and 19.9 K, respectively, for $x = 0.3$; and 20.9 K and 20.0 K, respectively, for $x = 0.4$. Compared with the results for bulk samples, a drastic enhancement of T_c is observed in these $\text{FeSe}_{1-x}\text{Te}_x$ films. Surprisingly, the values of T_c^{zero} in the films with $x = 0.2$ and 0.4 exceed 20 K. These values are larger than those reported for $\text{FeSe}_{0.5}\text{Te}_{0.5}$ films[14, 8, 10, 11]. In particular, the T_c of the $\text{FeSe}_{0.8}\text{Te}_{0.2}$ film with a thickness of 73 nm is approximately 1.5 times as high as those of bulk crystals of $\text{FeSe}_{1-x}\text{Te}_x$ with the optimal composition, $x \approx 0.5$ [7]. Based on the measurement of the ρ of the $\text{FeSe}_{0.8}\text{Te}_{0.2}$ film under a magnetic field applied along the c axis, we estimate an upper critical field at 0 K of $\mu_0 H_{c2} = 55.4$ T using the Werthamer-Helfand-Hohenberg(WHH) theory[16], which yields a Ginzburg-Landau coherence length at 0 K of

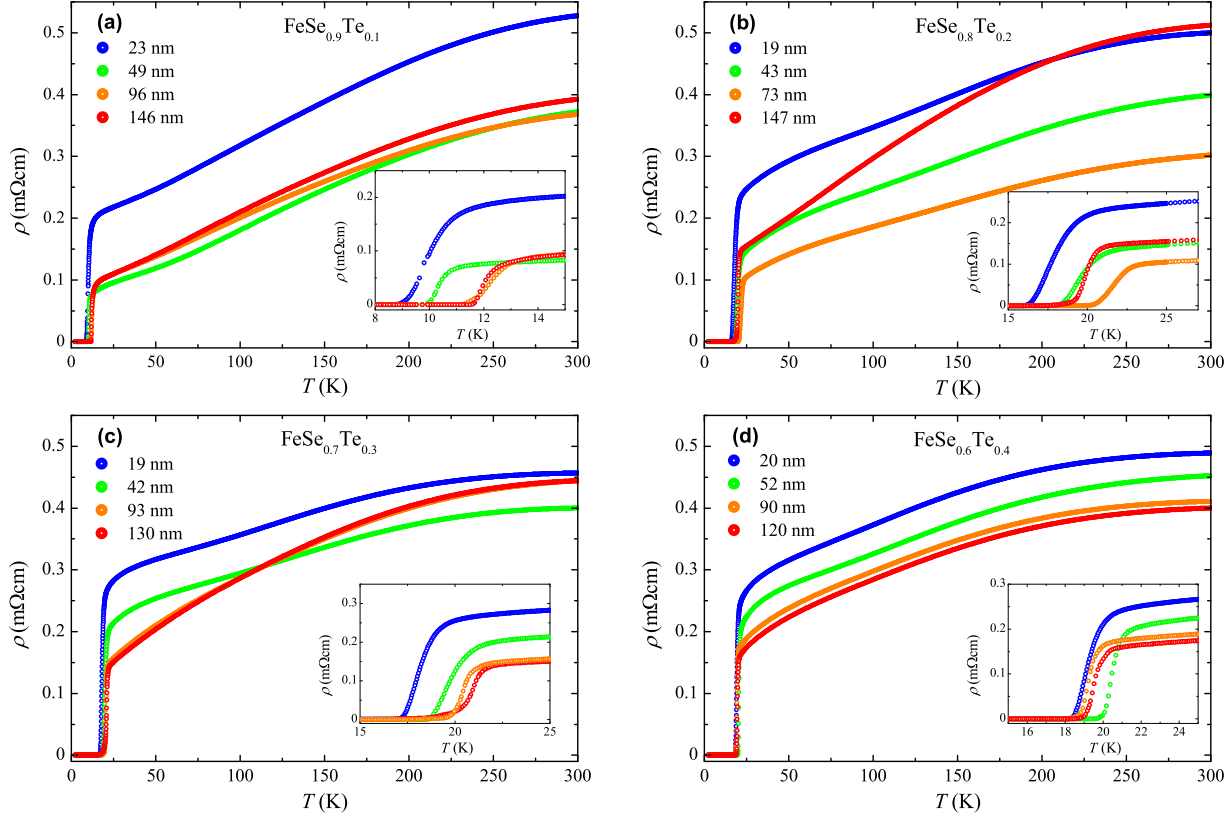


Fig. 2. Temperature dependences of the electrical resistivities, ρ , of FeSe_{1-x}Te_x thin films for (a) $x = 0.1$, (b) $x = 0.2$, (c) $x = 0.3$ and (d) $x = 0.4$ with different film thicknesses. The insets present enlarged views of the plots near the superconducting transition.

$\xi_{ab}(0) \sim 24.4$ Å (see the supporting information). This value of $\mu_0 H_{c2}$ is approximately half the value for an FeSe_{0.5}Te_{0.5} film on CaF₂ with a T_c of approximately 16 K.[9]

Using the data shown above, we present the phase diagram of FeSe_{1-x}Te_x films on CaF₂ substrates in Fig. 3. For comparison, the data for bulk samples of FeSe_{1-x}Te_x[7, 17] are also plotted in this figure. In bulk crystals, the optimal Te content to achieve the highest T_c is considered to be $x \approx 0.5$, and phase separation occurs in the region of $0.1 \leq x \leq 0.4$ [7]. However, our data clearly demonstrate that this phase separation is absent and that the optimal composition for an FeSe_{1-x}Te_x film on a CaF₂ substrate is not $x \approx 0.5$ but $x \approx 0.2$. It should be noted that the dependence of T_c on x suddenly changes at the boundary defined by $0.1 < x < 0.2$. Unlike the “dome-shaped” phase diagram that is familiar in iron-based superconductors, the values of T_c in films with $0.2 \leq x \leq 1$ increase with decreasing x , while the strong suppression of T_c is observed at $0.1 < x < 0.2$. The behavior in films with $x \geq 0.2$ can be explained by the empirical law which shows the relation between T_c and structural parameters. In iron-based superconductors, it is well accepted that the bond angle of (Pn, Ch)-Fe-(Pn, Ch) ($Pn = Pnictogen$, $Ch = Chalcogen$), α , [18, 19] and/or the anion height from the iron plane, h , [20] are the critical structural parameters that determine the value of T_c . In bulk samples of FeSe_{1-x}Te_x, α and h approach their optimal values, i.e. $\alpha = 109.47^\circ$ [18, 19] and $h = 1.38$ Å [20], with decreasing x (down to $x = 0$), which should be the same in FeSe_{1-x}Te_x films. Therefore, the increase of T_c in films with $0.2 \leq x \leq 1$ with decreasing x can be explained by the optimization of α and/or h based on the empirical law. However, the sudden suppression of T_c in films with $0 \leq x < 0.2$ is not consistent

with this scenario, and its origin should be sought among other factors. We consider there are two candidates for this origin from the structural analysis of bulk samples of FeSe_{1-x}Te_x. One is the effect of the orthorhombic distortion. In a bulk sample of FeSe, a structural phase transition from tetragonal to orthorhombic occurs at 90 K [21]. However, in bulk samples of FeSe_{1-x}Te_x with $x \sim 0.4 - 0.6$ which T_c s take optimum values, there are papers with different conclusions on the presence/absence of the similar type of the structural transition as that of FeSe [22, 23, 24]. It should be noted that a structural transition temperature is lower and that the orthorhombicity is much smaller than those of FeSe even in the report where the structural transition is present [24]. These results on crystal structures suggest that the orthorhombic distortion results in a suppression of T_c . This scenario is applicable to the behavior of our films, if a large orthorhombic distortion is observed only in films with $x = 0 - 0.1$. The other candidate is the change in the distance between the layers of Fe-*Ch* tetrahedra, δ . As shown in the supporting information, in polycrystalline samples of FeSe_{1-x}Te_x, the δ value of FeSe is much smaller than those of FeSe_{1-x}Te_x with $x \geq 0.5$ at which δ is nearly independent of x [22]. We speculate that the decrease of δ in FeSe is related with the suppression of T_c . Indeed, in polycrystalline samples, FeSe exhibits smaller values of δ and T_c than does FeSe_{0.5}Te_{0.5} [7, 22], and the intercalation of alkali metals and alkaline earths into FeSe result in the c -axis length as large as approximately 20 Å and T_c as high as 45 K [25, 26]. At this moment, the origin of the suppression of T_c at $0.1 < x < 0.2$ is unclear, which will be a subject of a future study. Regardless of its origin, we believe that it is reasonable to distinguish between superconductivity in $x = 0 - 0.1$ and in $x \geq 0.2$. In other words, our phase diagram of Fig. 3 provides a new view

for superconductivity in $\text{FeSe}_{1-x}\text{Te}_x$, that is, a discontinuity in superconductivity of $\text{FeSe}_{1-x}\text{Te}_x$. We are able to come to this picture, only after the data for $x = 0.1-0.4$ become available in this study. If we remove a cause for the suppression of T_c in $x = 0, 0.1$ in some way, a further increase in T_c can be expected because of the optimization of structural parameters.

In conclusion, we prepared high-quality epitaxial thin films of $\text{FeSe}_{1-x}\text{Te}_x$ on CaF_2 substrates using the pulsed laser deposition method. We successfully obtained $\text{FeSe}_{1-x}\text{Te}_x$ films with $0.1 \leq x \leq 0.4$, which has long considered to be the “phase-separation region”, using a thermodynamically non-equilibrium growth of film deposition. From the results of electrical resistivity measurements, a complete phase diagram is presented in this system, in which the maximum value of T_c is as high as 23 K at $x = 0.2$. Surprisingly, a sudden suppression of T_c is observed at $0.1 < x < 0.2$, while T_c increases with decreasing x for $0.2 \leq x < 1$. This behavior is different from the “dome-shaped” phase diagram that is familiar in iron-based superconductors.

Materials and Methods

All of the $\text{FeSe}_{1-x}\text{Te}_x$ ($x = 0-1$) films in this study were grown by the pulsed laser deposition method using a KrF laser [27, 28]. Polycrystalline pellets with a nominal composition of $\text{FeSe}_{1-x}\text{Te}_x$ ($x = 0-1$) were used as the targets. The substrate temperature, repetition rate, and back pressure were 280 °C, 20 Hz, and 10^{-7} Torr, respectively. Single crystals of the CaF_2 (100), which is one of the most preferred materials for the thin-film growth of $\text{FeSe}_{1-x}\text{Te}_x$ [9, 4], were used as the substrates. We used a metal mask to prepare the $\text{FeSe}_{1-x}\text{Te}_x$ films in a six-terminal shape for transport measurements. The thicknesses of the thin films were measured using a Dektak 6 M stylus profiler and estimated to be 12-148 nm. The crystal structures and orientations of the films were characterized by four-circle X-ray diffraction (XRD) with $\text{Cu K}\alpha$ radiation at room temperature. The a -axis and c -axis lengths are determined from the 204 and 00 l reflections in XRD measurements, respectively. Electrical-

resistivity measurements were conducted using the four-terminal method from 2 K to 300 K with magnetic fields up to 9 T applied perpendicular to the film surface.

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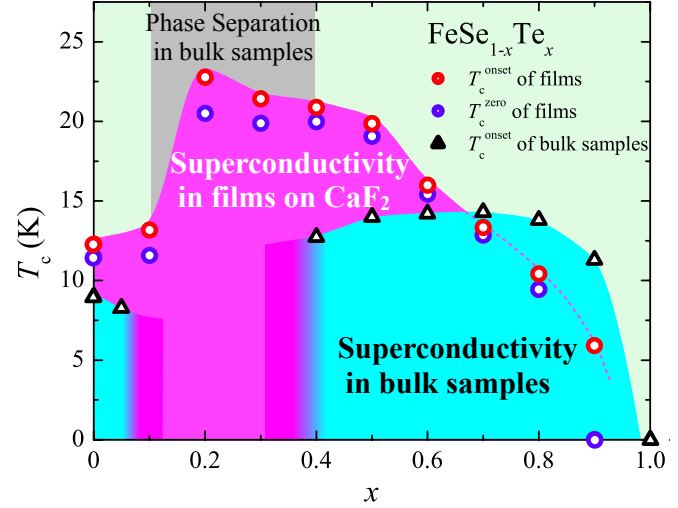


Fig. 3. Dependence of T_c on x . The red and blue circles represent the T_c^{onset} and T_c^{zero} values of the $\text{FeSe}_{1-x}\text{Te}_x$ thin films, respectively. The black triangles represent the T_c^{onset} values obtained in measurements of the magnetic susceptibility of bulk samples [7, 17]. The dashed curve is a guide for the eye.

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